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Indian Standard

TEST METHOD FOR OXIDATION STABILITY OF INHIBITED MINERAL INSULATING OILS

UDC 621·315·615·2 : 543·872

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NEW DELHI 110002

Gr 4 *September* 1989

Indian Standard

TEST METHOD FOR OXIDATION STABILITY OF INHIBITED MINERAL INSULATING OILS

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 25 May 1988, after the draft finalized by the Liquids and Gaseous Dielectrics Sectional Committee had been approved by the Electrotechnical Division Council.

0.2 This standard deals with the test method for the determination of oxidation stability of unused inhibited mineral insulating oils. This standard shall be used in conjunction with IS : 335 - 1983*.

0.3 Oxidation of insulating oils due to the formation of gases, deteriorates the electrical properties of insulating materials considerably. It is necessary to determine the stability of such oxidized oils to evaluate their performance as insulating medium, when being used in the long range. The test method specified in this standard determines the oxidation stability of unused inhibited insulating mineral oils by measuring the induction period. This method can also be used to obtain information on the acid and sludge forming tendency before or after the induction period.

0.4 The continuity of oil supplies can be tested by this method. This test method does not ensure or provide any information about the oil, when used in service. As such any information about the performance of the oil during service is not within the scope of this standard.

0.5 In the preparation of this standard, considerable assistance has been taken from IEC standard 474 (1977) 'Test method for oxidation stability of inhibited mineral insulating oils', issued by the International Electrotechnical Commission.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2 - 1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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*Specification for new insulating oils (*third revision*).

*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard deals with the test method for determination of oxidation stability of unused inhibited mineral insulating oils. The results of this test method do not provide any information of the performance of the oil under service conditions.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply, in addition to those given in IS : 335 - 1983*.

2.1 Induction Period — Induction period is the time taken for the oil to develop a volatile acidity corresponding to a NV (VA) = 0·28 mg KOH/g oil.

2.2 Repeatability — A quantitative measure of the variability associated with a single operator in a given laboratory obtaining successive repeat results on the same apparatus. It is defined as that difference between two such single results that would only be exceeded in the long run in one case in twenty in the normal and correct operation of the test method.

2.3 Reproducibility — A quantitative measure of variability associated with operators working in different laboratories, each obtaining single result on identical test material. It is defined as that difference between two such single and independent test results that would be exceeded in the long run only in one case in the normal and correct operation of the test method.

3. TEST METHOD

3.1 General — The time required for an oil to develop a volatile acidity corresponding to a neutralization value NV (VA) of 0·28 mg KOH/g oil is measured. The oil sample is maintained at 120°C in the presence of a copper catalyst coil while bubbling through a constant flow of oxygen. Volatile oxidation products are

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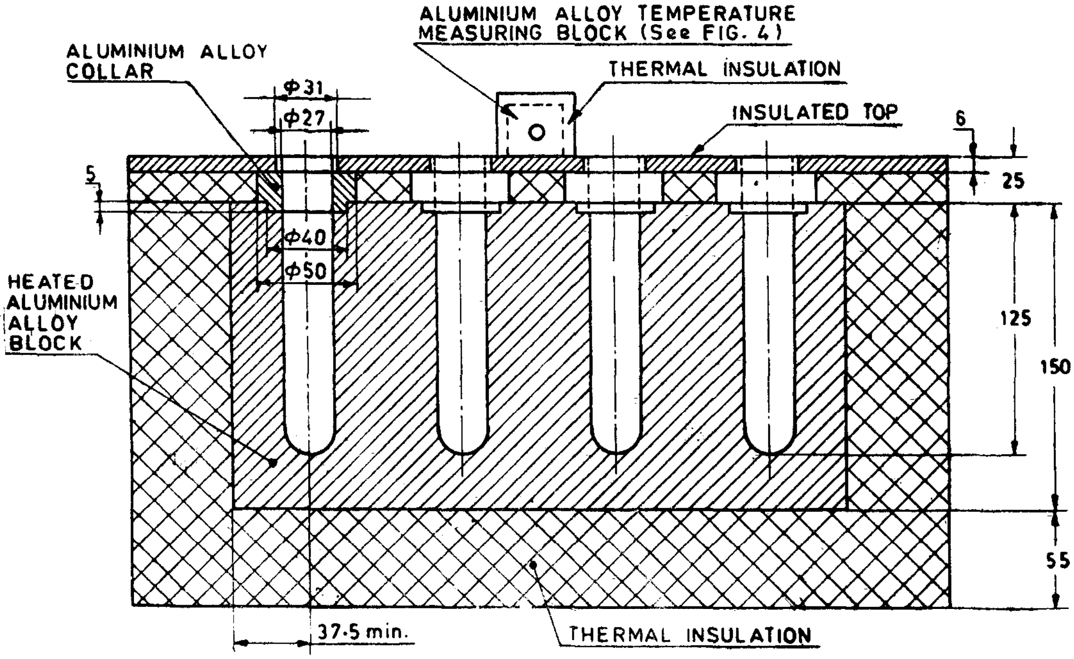
*Specification for new insulating oils (*third revision*).

observed in aqueous alkali containing phenolphthalein. The completion of the test is indicated by the discolouration of the absorbing solution.

3.2 Test Equipment

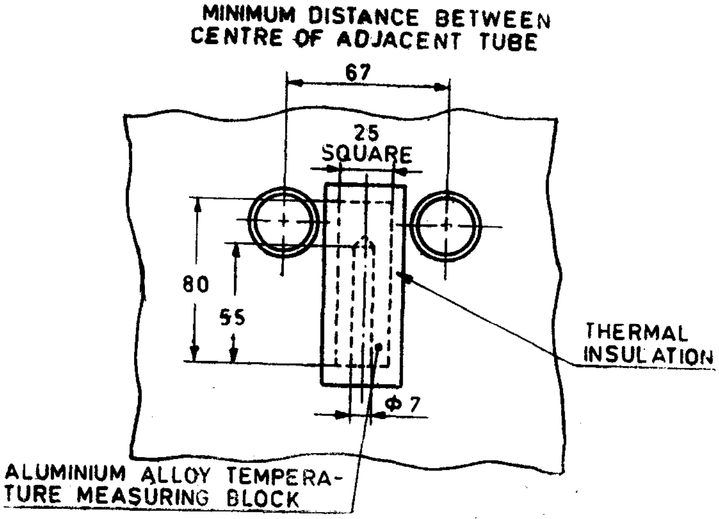
3.2.1 *Heating Arrangement*

3.2.1.1 A thermostatically controlled aluminium alloy block heater or oil bath may be used to maintain the oil in the desired number of oxidation tubes at the required temperature of 120 ± 0·5°C (*see* Fig. 1 and 3). This temperature shall be read on a thermometer inserted in an oxidation tube to within 5 mm from the bottom; this oxidation tube shall be filled with oil up to the immersion line of the thermometer and placed in the heating bath. The temperature of the upper surface shall be maintained at 60 ± 5°C. Measure this temperature by the use of a thermometer in a drilled aluminium block (*see* Fig. 2). The surfaces of this block, other than that against the upper surface of the heating device, are protected by suitable insulation, for example, asbestos sheets of 4 mm thickness. This block should be placed as near to the holes as practicable and within the area of the upper surface covering the heating device.



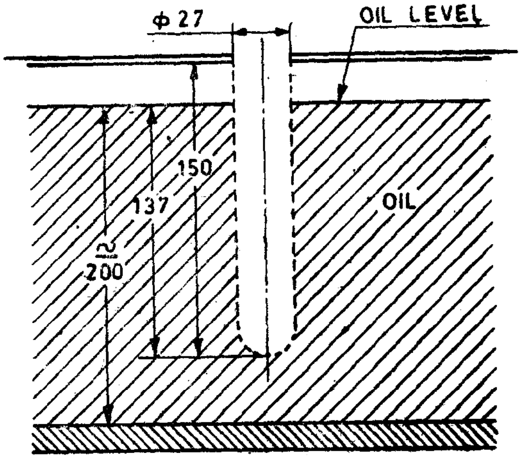
All dimensions in millimetres.

FIG. 1 TYPICAL 8 HOLES (4×2) ALUMINIUM HEATING BLOCK


MINIMUM DISTANCE BETWEEN CENTRE OF ADJACENT TUBE
67
25 SQUARE
80
55
Φ7
THERMAL INSULATION
ALUMINIUM ALLOY TEMPERATURE MEASURING BLOCK

All dimensions in millimetres.

FIG. 2 ALUMINIUM ALLOY TEMPERATURE MEASURING BLOCK



All dimensions in millimetres.

FIG. 3 POSITION OF THE TUBE IN THE OIL BATH

3.2.1.2 When using an aluminium heating block, the oxidation tubes are inserted into the holes to an overall depth of 150 mm. The depth of the holes in the heating part of the block shall be at least 125 mm and short metal collars, passing through the insulating cover and surrounding each oxidation tube, will ensure heating over the 150 mm length of the tube.

3.2.1.3 In the case of oil baths, the oxidation tubes shall be immersed to a depth of 137 mm in the oil and to an overall depth of 150 mm in the bath (*see* Fig. 3).

3.2.1.4 For both types of heating device, the height of the oxidation tubes above the upper surface shall be 60 mm and the diameter of the holes shall be just sufficient to allow insertion of the specified tube. In the case of slackness, a 25 mm diameter O-ring may be placed round the tube and pressed against the insulated top. The heating bath should be equipped with supports to hold the absorption tubes.

NOTE — The test shall be conducted away from sun light.

3.2.2 *Test Vessels*

These consist of borosilicate or neutral glass test tubes provided with a B 24 ground joint of the following dimensions. The test tube is fitted with a Drechsel head to which is attached the inlet tube which extends to within 2·5 ± 0·5 mm from the bottom and has its end ground at an angle of 60° to the horizontal axis (*see* Fig. 4).

a) Overall length, mm	210 ± 2
b) External diameter, mm	26 ± 0·5
c) Wall thickness, mm	1·4 ± 0·2
d) Height of the head, mm	28 ± 2
e) Oxygen inlet tube :	
1) External diameter, mm	5·0 ± 0·4
2) Wall thickness, mm	0·8 ± 0·1

3.2.3 *Absorption Tubes*

These are identical with the test vessels and the distance between axis of the two tubes shall be 150 ± 50 mm Connections between the test and absorption tubes should be as short as possible, of glass tubing butt jointed to the vessels by means of short flexible sleeves (silicon rubber sleeving has been found suitable for this purpose). These tubes are mounted outside the heating device.

3.2.4 *Filtering Crucibles* — Gooch-type crucibles with fused-in fitted glass disk of grade 4 porosity (5 to 15 microns), for example, of 35 ml capacity.

3.2.5 *Determination of the Maximum Diameter of the Pores of a Glass Filter*

3.2.5.1 The determination of the maximum diameter of the pores provides an indication of the retaining capacity of the filter, that is the maximum diameter of spherical particle that can pass through it.

3.2.5 2 The maximum diameter of the pores is determined by measuring the air pressure necessary to cause the first air bubble to pass through the filter moistened by the liquid to be tested.

3.2.5.3 The following relationship is used:

$$D = \frac{400y}{P}$$

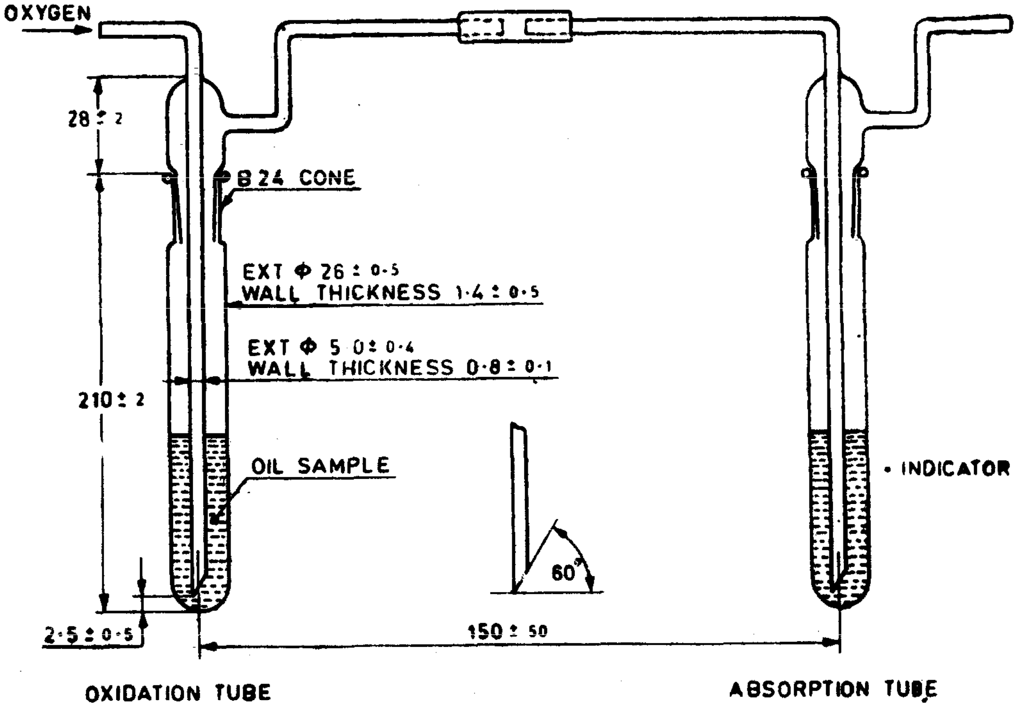
where

D = diameter, in micrometres, of the pore;

y = surface tension of the test liquid, in Newtons/metre; and

P = observed pressure, in Pascals.

NOTE — In order to check a filter having a maximum pore diameter of 4 to 10 µm, either water or carbon tetrachloride is used as a test liquid.



All dimensions in millimetres.

FIG. 4 TEST VESSEL AND ABSORPTION TUBE

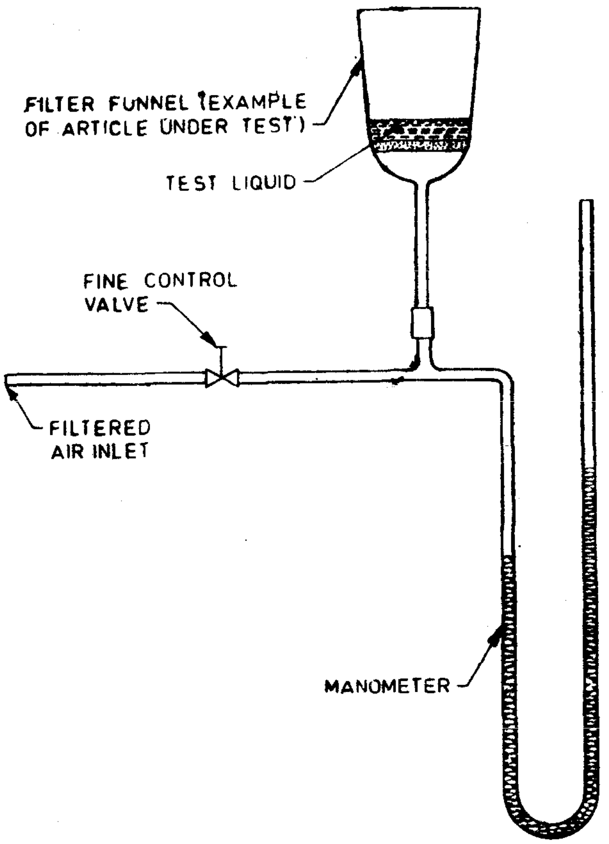


FIG 5 APPARATUS FOR DETERMINATION OF MAXIMUM DIAMETER OF THE PORES OF A GLASS FILTER

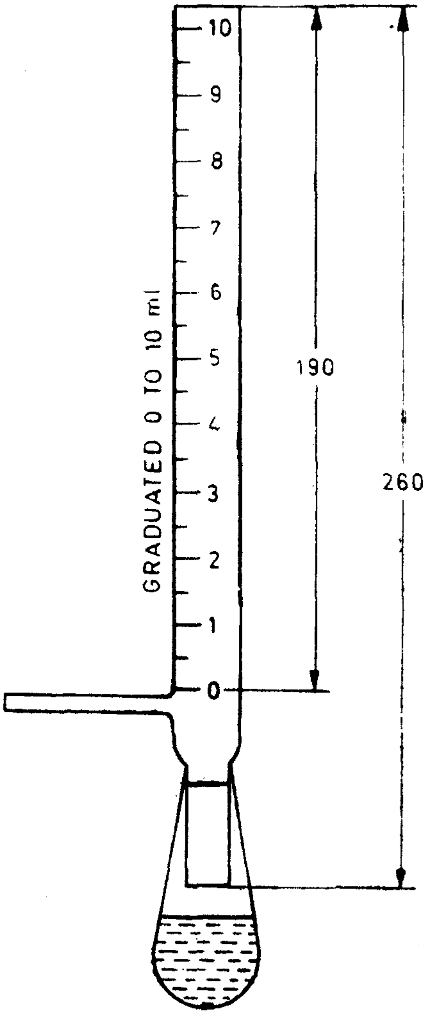
3.2.5.4 The washed filter is completely immersed in the test liquid of which a film of several millimetres thickness will remain on the upper surface of the filter attached to the apparatus (*see* Fig. 5).

3.2.5.5 A uniformly increasing pressure is applied to the filter until one or two bubbles of air appear in the liquid above the filter. The manometer reading enables the maximum diameter of the pores to be calculated. This manometer can be directly calibrated in pore diameters.

3.2.5.6 For a filter, whose maximum pore diameter is between 4 and 10 μm, the manometer reading in Pascals will be 72 to 29 kPa for water and 26 to 10·5 kPa for carbon tetrachloride (*see* Fig. 5).

3.2.6 *Porcelain Vessels* — Capacity : 50 ml.

3.2.7 *Soapmeter* — For checking oxygen flow rate (*see* Fig. 6).



All dimensions in millimetres.

FIG. 6 RUBBER RESERVOIR CONTAINING SOAP SOLUTION

3.2.8 *Timer* — For checking oxygen flow rate Subdivisions of the graduation 0·2 s.

3.2.9 *Oxygen Supply Train* — Oxygen should be dried by passing through concentrated sulphuric acid and then through a drying tower filled with glass wool and soda asbestos. Any other method known to be of equal or greater efficiency can, however, be used. Oxygen is then metered into each test tube by any suitable gas flow measuring device.

3.2.10 *Burette* — Volume 10 ml with graduations of 0·01 ml.

3.2.11 *Thermometer* — A thermometer conforming to the requirements given in Appendix A.

4. REAGENTS

4.1 Alkali Blue 6 B Indicator — 2 g of alkali blue 6 B per 100 ml of azeotropic ethanol (about 5 percent water). Extract 2 g of alkali blue 6 B with azeotropic ethanol in a Soxhlet apparatus, filter, if necessary, and dilute to 100 ml with azeotropic ethanol. Add 3 ml of 0·1 N HCl and after 24 hours carry out a check acid value to confirm that the indicator has been sensitized, that is a colour change. Should sensitization not be sufficient, then add more 0·1 NHCl until sensitization has occurred. Periodic sensitization may be necessary.

4.2 Phenolphthalein Indicator — 1 g of phenolphthalein per 100 ml of azeotropic ethanol (about 5 percent water).

4.3 Titration Solvent — Mix 3 parts by volume of toluene (sulphur-free grade) and 2 parts by volume of azeotropic ethanol.

4.4 Potassium Hydroxide — 0·1 N alcoholic solution.

4.5 Oxygen — Obtained from liquid air, 99·4 percent minimum purity.

4.6 Hydrochloric Acid — Aqueous 0·1 N solution.

5. PREPARATION OF GLASSWARE

5.1 Method of Cleaning the Test Vessels

5.2 The test tube shall be chemically cleaned. A method of cleaning, which has been found satisfactory, is to wash with acetone, distilled water, then with concentrated sulphuric acid which is removed by washing, first with tap water, then with distilled water.

5.3 The reagents used shall be of chemically pure grade.

5.4 The apparatus is dried in an air-oven at 105-110°C for at least 3 hours and then allowed to cool to room temperature in a desiccator in which the test-tubes are kept until they are used.

6. CATALYST

6.1 A 900 mm length of soft electrolytic copper wire is rolled into a spiral approximately 20 mm external diameter.

6.2 Immediately before use, the requisite length of copper wire is cleaned with 100 grade silicon carbide abrasive cloth. All traces of abrasive are removed with a lintless filter paper and then with a dry lintless cloth.

6.3 Roll the wire into a spiral of approximately 2 cm external diameter and 5 cm long.

6.4 The spiral is thoroughly cleaned by dipping it into normal heptane, then dried in air and immediately introduced into the test vessel.

6.5 To avoid contamination, the prepared copper shall be handled only with tweezers. The copper wire shall not be re-used.

7. OIL SAMPLE CONDITIONING

7.1 Oil to be tested shall be filtered through a previously dried (1 h at 105°C) fritted glass filter (grade 4 porosity) to remove traces of sediment, fibre and excess water. The first 25 ml of filtrate should be discarded.

8. PROCEDURE

8.1 Preparation of the Test — Adjust the heating bath to maintain the oil in the desired number of oxidation tubes at the required temperature of 120 ± 0·5°C. Weigh 25 ± 0 1 g of oil into the oxidation tube and insert the catalyst coil prepared as described in **6.** Insert the Drechsel head and place the tube into the heater. Pour into the absorption tube, 25 ml of an alkaline solution prepared by diluting 50 ml of the 0·1 N alcoholic KOH solution to 1 litre with distilled water. Add a few drops of phenolphthalein indicator solution. Insert the Drechsel head and connect to the oxidation tube Adjust the oxygen flow to deliver 1 ± 0·1 l/h measured by means of the soapmeter connected to the outlet end of the absorption tube (*see* Fig. 4).

8.2 Oxidation

8.2.1 Oxidize the oil while maintaining its temperature at 120 + 0·5°C and an oxygen flow rate of 1 ± 0·1 l/h. Check oxygen flow and temperature daily. Twice a day (at the beginning and end of the working day), inspect the absorption tube for discolouration.

Note the time at which discolouration is observed and discontinue the test then or after 236 h, whichever period is the shorter.

8.2.2 If required, determine after a specified duration, additional characteristics (sludge content, soluble, volatile and total acidities, oxidation rate) of the oxidized oil according to **8.4**.

NOTE — Phenolphthalein fades rather quickly when exposed to strong direct light. Should a faint tint be observed, it is suggested that a few more drops of indicator are added.

8.3 Determination of the Induction Period — For the purpose of this method, the induction period is arbitrarily set as the time taken for the oil to develop a volatile acidity corresponding to a NV (VA) = 0·28 mg KOH/g oil. Report, as induction period, the mean of the times corresponding to the two visual inspections immediately preceding and following discolouration of the absorption tube. The period between the two final inspections should not be greater than 20 h.

NOTE 1 — When following the above procedure, the interval between two consecutive inspections is 8 h during the day and 16 h at night. In order to reduce this interval, two tests should be run, the first being started, for instance, at 0900 h and the second at 1 00 h. This schedule of tests offers a series of inspections spaced 8 h apart without exceeding the limits of an 8 h day.

When no information is available on the test duration, a new set of the tests should be started two days later to avoid extending the inspections through the week-end.

NOTE 2 — Induction periods can be more precisely and automatically measured by the break in the curve obtained by continuously recording the *p*H of the aqueous alkaline absorbent. In this case absorption tubes need to be modified.

8.3.1 The provisions of Appendix B of IS : 12177-1987* shall apply.

8.4 Volatile Acidity Determination — Titrate the water in the absorption tube using 0·1 N alcoholic KOH solution and calculate as in **8.4.2.** Add to the result obtained, 0·28 mg KOH/g oil to obtain the volatile acidity formed during the total test period.

8.4.1 *Total Acidity* — The total acidity is obtained by adding the soluble acidity to the volatile acidity.

8.4.2 *Oxidation Rate Measurement*— It wil sometimes be useful to obtain the oil oxidation rate over the entire test period. A graphical representation of this can be obtained if volatile acidity is daily titrated and the NV (VA) plotted against time. In this case, the induction period is obtained by the time corresponding to the NV (VA) = 0·28 mg KOH/g oil. Pure water (25 ml) is used as the absorbing agent instead of the alkali solution. The total volatile acidity NV (VA) is the sum of the daily volatile acidities.

For daily titrations, the following procedure is adopted:

a) Disconnect the absorption tubes;

b) Titrate the volatile acidity with 0·1 N alcoholic KOH solution using a few drops of phenolphthalein as indicator;

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*Methods of test for oxidative ageing of electrical insulation of petroleum oils by the open beaker method.

c) Reconnect the absorption tube without replacing the water; and

d) Calculate the volatile neutralization value, in milligrams of KOH per gram of oil, as follows:

$$\text{Daily volatile acidity} = \frac{N \times 56{\cdot}1 \times a}{25}$$

where

N = normality of the alcoholic KOH solution, and

a = ml of 0·1 N KOH alcoholic solution used in the titration.

9. NUMBER OF TESTS

9.1 The test shall be done in duplicate.

10. REPORT

10.1 The average of the two determinations is reported as the induction period.

11. PRECISION

11.1 The following criteria should be used for judging the acceptability of results (95 percent confidence):

Repeatability (r) : Duplicate results by the same operator should be considered suspect, if they differ by more than the amount mentioned in the table hereunder.

Reproducibility (R) : The results submitted by each of two laboratories should be considered suspect, if the two results differ by more than the amount mentioned in the table hereunder:

Induction Period	100 h
(r)	(R)
10 percent	40 percent

12. LIMIT

12.1 Limits for the induction period, sludge and any of the other determination should be based on the performance of the oil of which the continuity in properties is being established.

12.2 For guidance only, it may be stated that, induction periods in the range of 120 to 200 h are typical.

APPENDIX A

(*Clause* 3.2.11)

THERMOMETER SPECIFICATION

Range	98 to 152°C
Immersion line from the bottom	100 mm
Graduations:	
Subdivisions	0·2°C
Long lines at each	1°C
Number at each	2°C
Scale error, *Max*	0·2°C
Expansion chamber permits heating to	180°C

Total length	395 ± 5 mm
Stem diameter	6·0 to 7·0 mm
Bulb length	15 to 20 mm
Bulb diameter	Not greater than stem
Distance from the bottom of bulb to 98°C	125 to 145 mm
Distance of contraction chamber to top, *Max*	35 mm

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